Laser Particle Size Analysis Instruction (CZO Project)

Compiled by Mohaddese Effati.

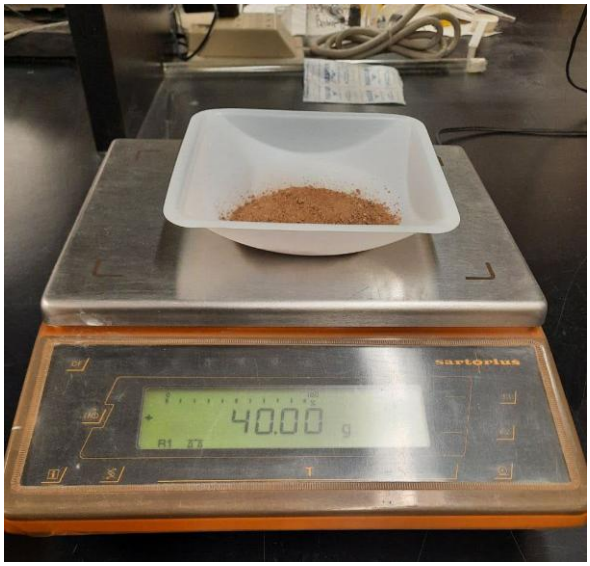
Edited by Xenia De Gracia for Mine Tailing samples (follow comments in pink)

1. ~~**Oven-drying:** place soil sample into the 105°C oven for 24 hours.~~ *Oven-dry at 35 Celsius or freeze-dry.*

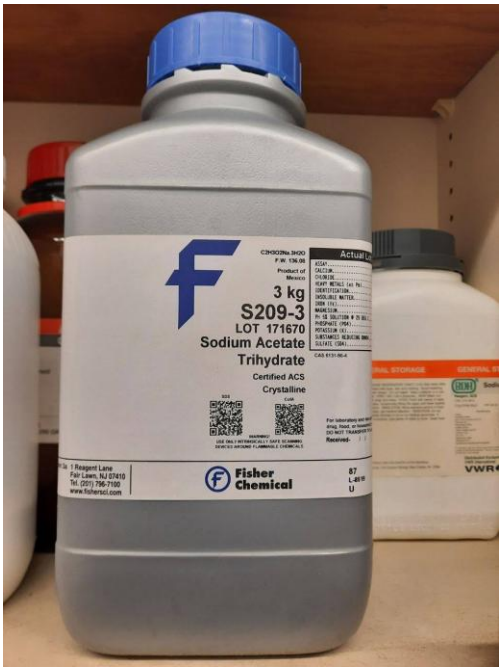
2. ~~**Sieving:** Use the sieving machine (dry sieving) for 5 minutes to separate particles larger than 2 mm.~~ *For tailings, we already sieved them with 2mm.*

3. Carbonate Removal:

- ~~Use 40.0 g of sieved soil sample and place it in a 250-ml centrifuge tube. Record your sample number.~~ *We don't want to remove the carbonates from our tailings.*



- ~~Add 10.0 ml of 1.0 M Sodium Acetate (pH 5.0) and 100 ml of deionized water on top of the soil in the centrifuge tube.~~

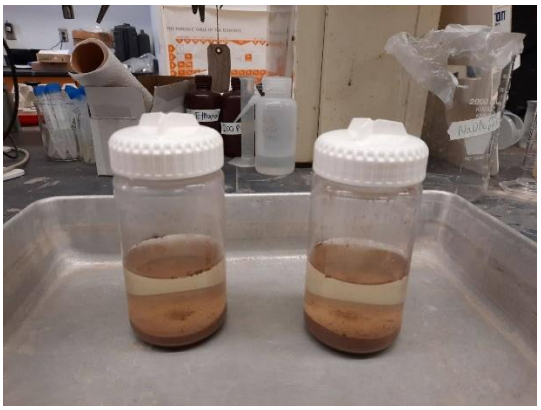


- ~~Tighten the tubes' cap and handshake bottles to mix.~~

- ~~Loosen caps to allow gas to escape, then place bottles in a hot water bath. Heat until bubbling stops (<30 min).~~

~~- Balance bottles for centrifuge; if not add a few drops of DI water to reach equal weights.~~

~~- Centrifuge for 15 min at 3600 rpm until the supernatant is clear.~~



~~- Check to see if there is no soil in suspension. If not, decant and add 50 ml of DI water.~~

- ~~Balance and handshake bottles to mix.~~
- ~~Centrifuge for 15 min at 3600 rpm until the supernatant is clear.~~
- ~~Wash one more time with 50 ml of deionized water.~~
- ~~Balance, mix, centrifuge, and decant supernatant.~~

~~4. Organic Matter Removal with Bleach.~~

- ~~Add 150 ml of bleach - NaOCl (pH 9.5) into the bottle~~. *Don't need this procedure because we don't have Organic Matter in mine tailings.*

- ~~Tighten cap and handshake bottles to mix.~~

- ~~Put the soil-bleach mixture in hot water bath (low heat setting) until bubbling stops.~~

- ~~Allow bottles to cool down, then balance bottles, centrifuge, and decant supernatant.~~

- ~~Repeat bleach process as needed if high in organic matter.~~

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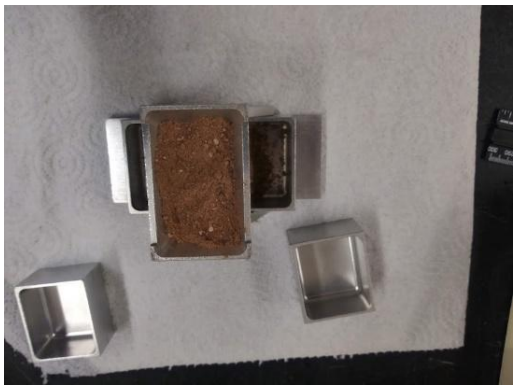
- ~~Add 100 ml of DI water, balance the bottles, then shake bottles to mix.~~

- ~~Centrifuge and decant supernatant.~~

- ~~Wash for 2nd time with 100 ml of DI water. If clays do not settle after being centrifuged, do not decant supernatant.~~

5. **Drying the soil Samples:** In order to dry the soil samples, after decanting clear supernatant for third time, they may be placed in the 40°C oven until they are completely dry.

6. **Using the Soil Splitter:** After crushing dried pretreated soils, use the soil splitter to have an evenly distributed soil particle. Use 10 grams of each sample and repeat the splitting at least 5 times. Use the remaining soil to weigh and use in laser diffractometer device.



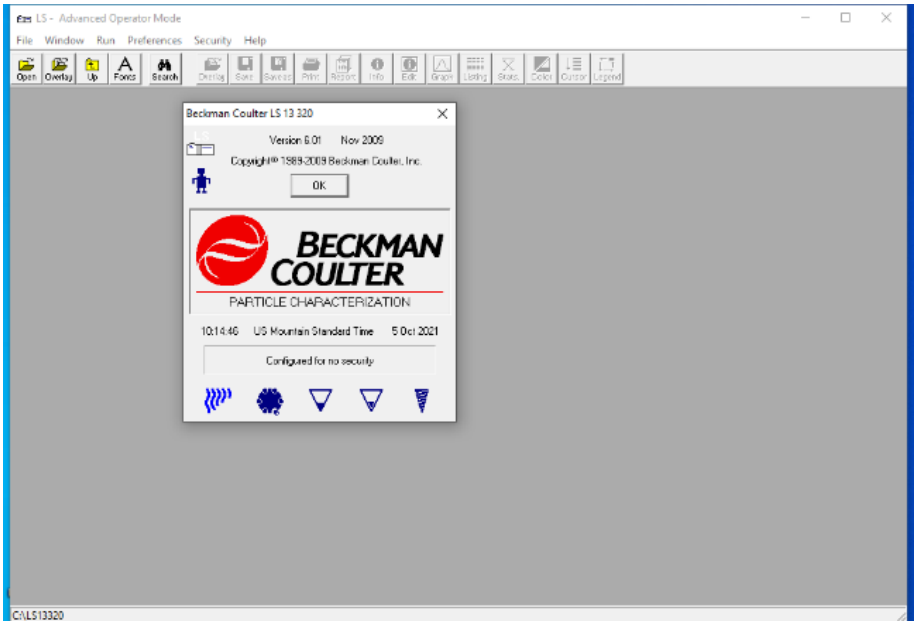
7. **Running the Obscuration test:** This is to determine the approximate amount of sample to weigh out for the actual particle size measurements.

- Turn on the fan located on the back of the Aqueous Liquid Module (The power switch is located under the bottle of dispersant on the back right of the machine).

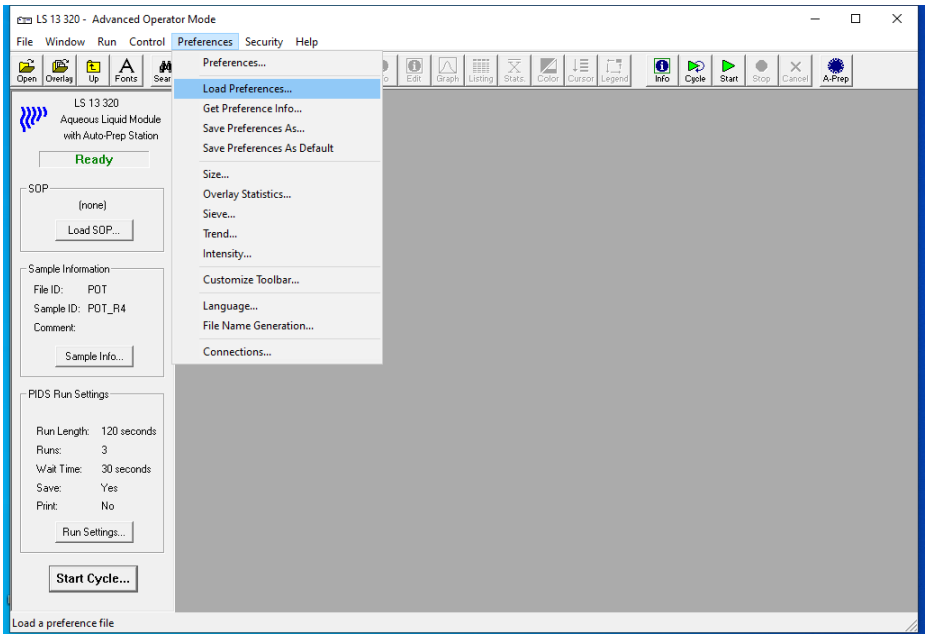
- Pick up the sonicator and slide it onto the mount located above the water dish.

- Log into the computer using the password “1980vanGenuchten” on the ”Tuller” account.

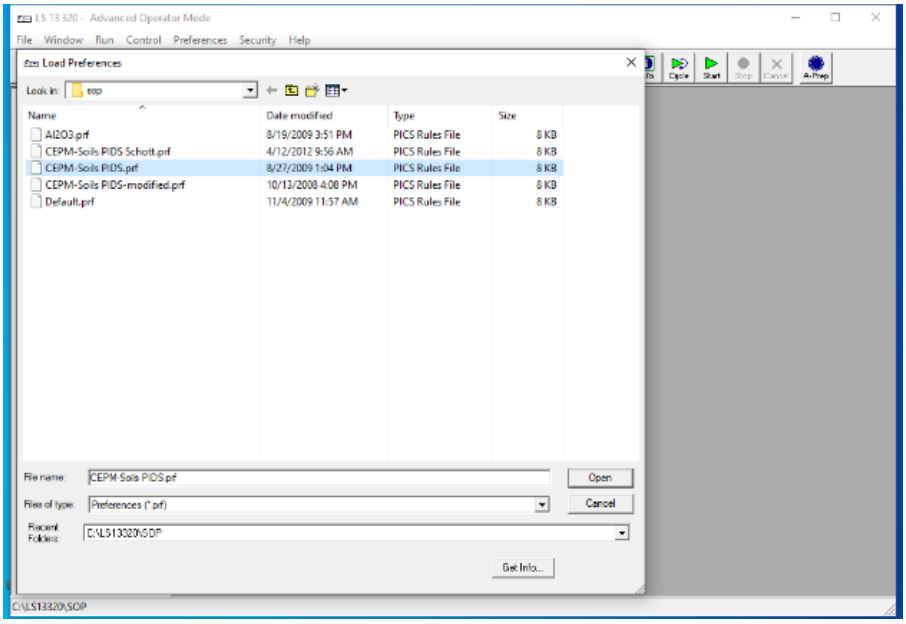
- Open the software on the computer. Hit “OK” to exit to the main screen.



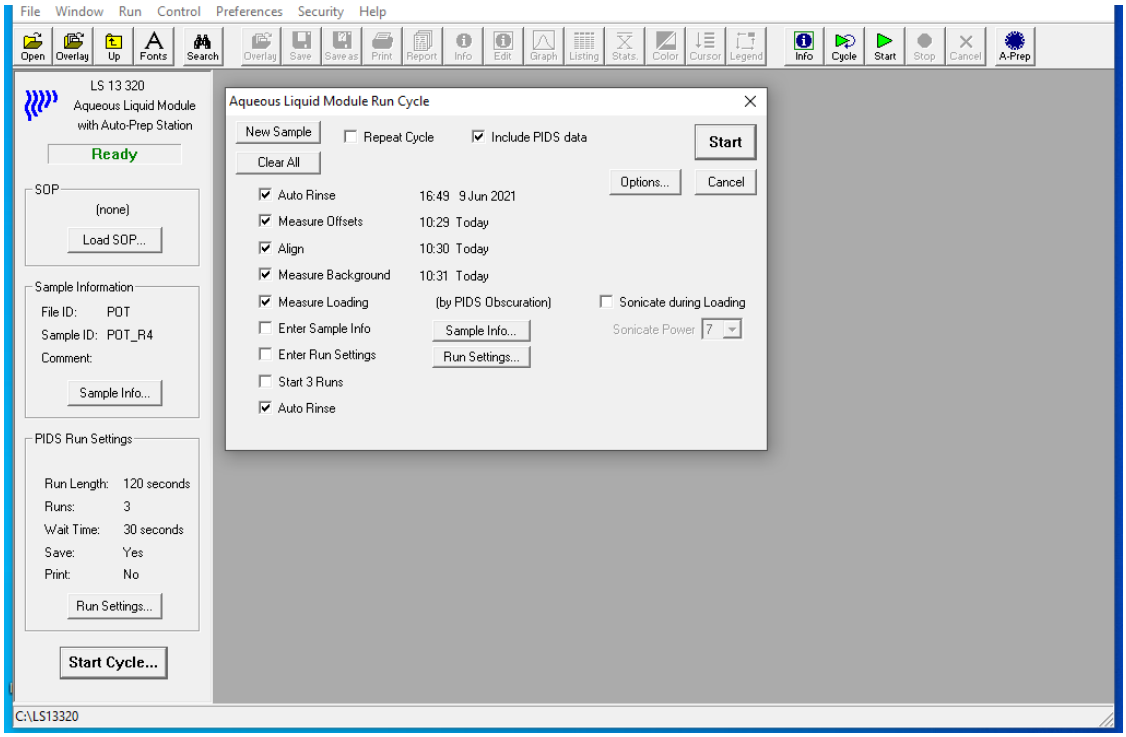
- Select “Preference” and the “Load Preferences”.



- Select the PIDS preference (the unmodified version).



- Select “Start Cycle” and beginning with the first “Auto Rinse”, check all the boxes through “Measure Loading”. Also select the final “Auto Rinse”. Make sure the box in top middle that says “Include PIDS data” is also selected. Then press “Start”.



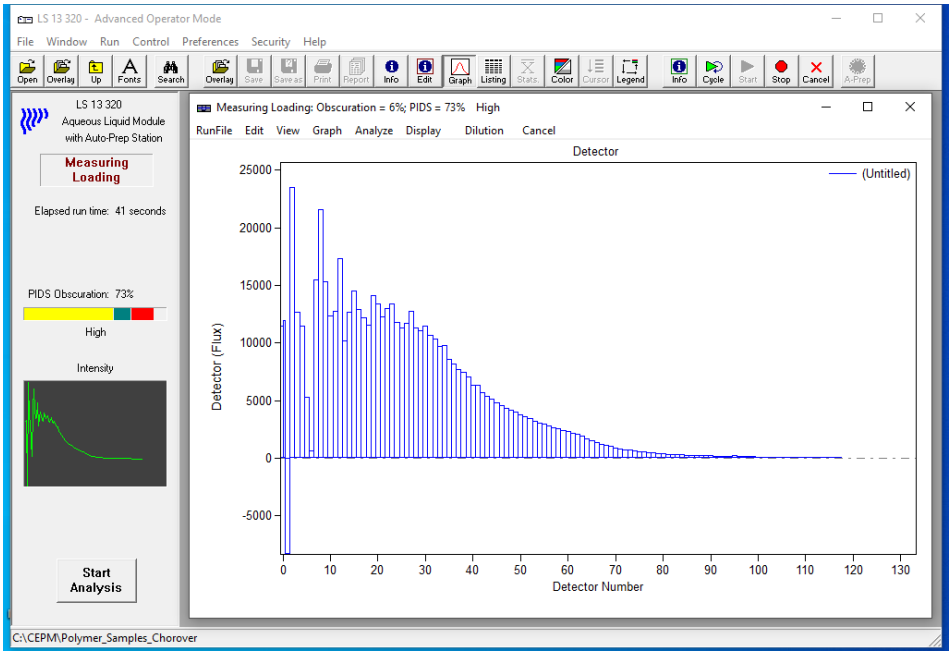
- Choose a soil sample, If the sample has a lot of clay/small particles, less sample will be needed to reach the desired obscuration range. Generally, a good starting point (except for very finely textured samples) is to weigh out 0.100 grams.

  More samples can always be added during the obscuration tests, so it is always better to start with less sample than looks to be necessary.

- Load your sample directly into the water dish once the computer screen reads “Measure Loading”.

- Before loading the sample, the obscuration for both the PIDS and Standard Obscuration should be approximately 0, and the screen should ask for additional sample.

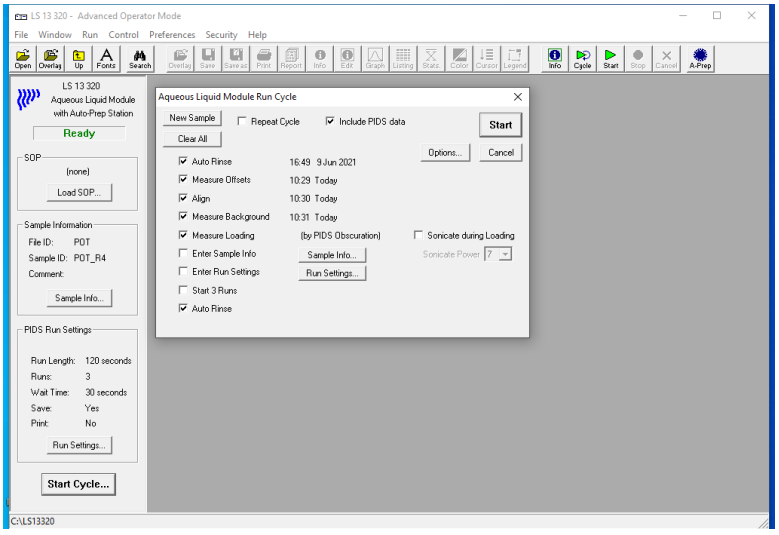
- Wait 30 seconds and look at the PIDS Obscuration value. Ideally, the Obscuration value at the time of running the sample should fall between 45-60%. Staying on the lower end of the 45-60% spectrum is preferred.

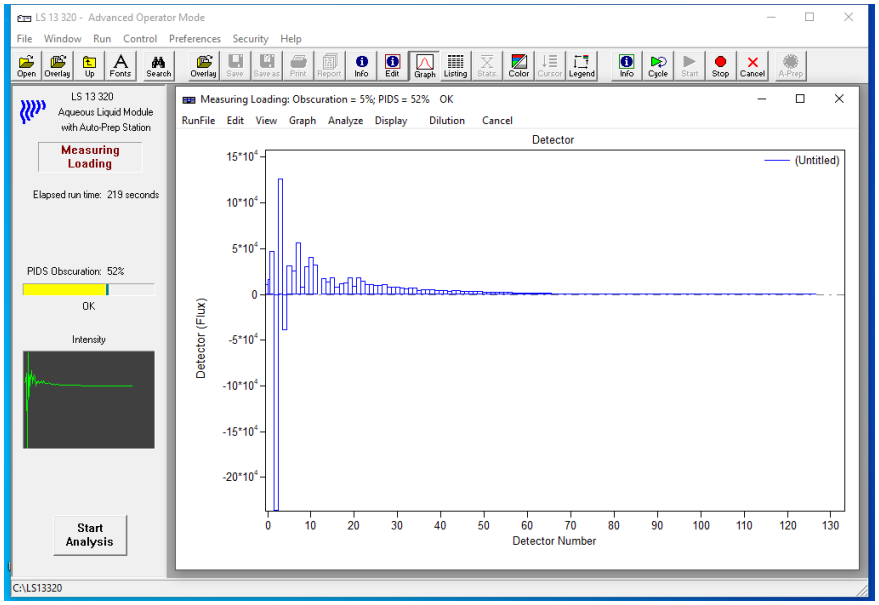


- If the obscuration value is too high select “Start Cycle” to Auto Rinse the system. This will eliminate the sample from the system.

- After the Auto Rinse is done, select “Start Cycle “, this time choosing only “Measure Loading” and “Auto Rinse” (the one at the bottom of the checklist). Measure out a smaller amount of sample to reach a lower obscuration value.

- Repeat this process with every sample available to run and record the weights required to reach the desired obscuration level.





- Since the obscuration value measured during this process occurs before the dispersion process with water and sodium hexametaphosphate, the obscuration value during the laser diffraction will most likely be a little higher than the value currently shown on the screen.

- if the obscuration value is too low then add more sample into the water dish until the PIDS obscuration value reads an appropriate value.

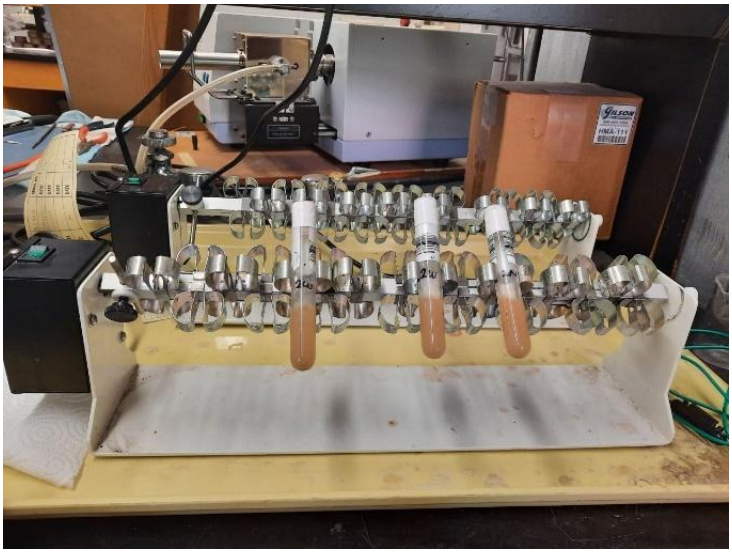
- If this process takes longer than 90 minutes or the obscuration values do not return to a low value after each Auto Rinse (between 0-5%) then the next time that you go to the “Start Cycle” option, select all of options through “Measure Loading” again (along with the final Auto Rinse). This allows for a readjustment of the system which keeps the obscuration values more accurate.

- Keep track of the amount of sample weighted into the dish. Record the amount of sample needed to reach the correct obscuration value and then hit the “Start Cycle” located in the bottom right-hand corner of the screen. That will start Auto Rinse which will rinse out the sample from the system.

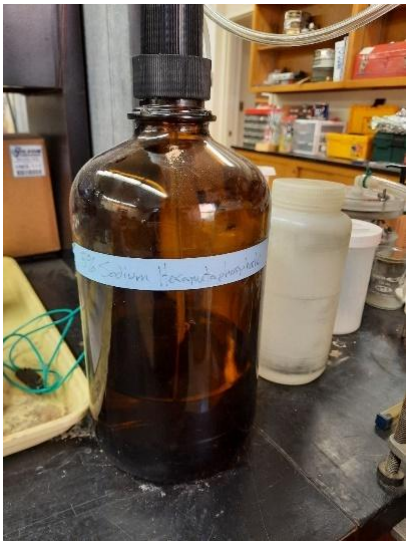
- At the conclusion of finding the obscuration values for all the samples do a final Auto Rinse. Then go to “Control” and select “Pump Off”.

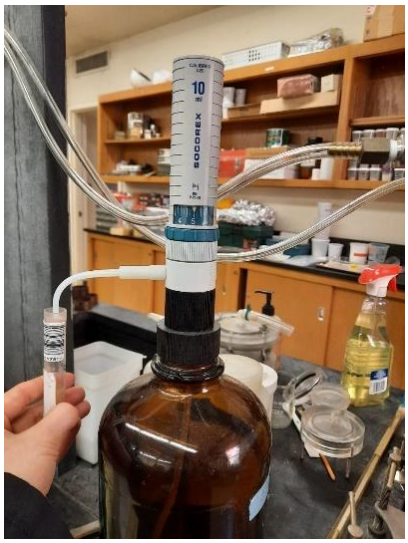
8. **Measuring the Samples:** Weigh your soil based on the desired weight which was determined in the obscuration test process (Step 7) and put them into tube. For each sample, three test tubes will be prepared to create replicants.

9. **Pretreatment of soil samples:** Once all samples are weighed out (28 tubes maximum per set), then slowly add 5 mL of water to each tube, put the cap on and make sure they are solidly attaching (preferably use the tape around the cap) and place them on the sample rotator for **24 hours**.



- **After 24 hours**, shake each of the tubes, remove the caps and add 5 mL of Sodium Hexametaphosphate to each of the tubes. Recap the tubes and place them on the rotator for a second 24 hours.



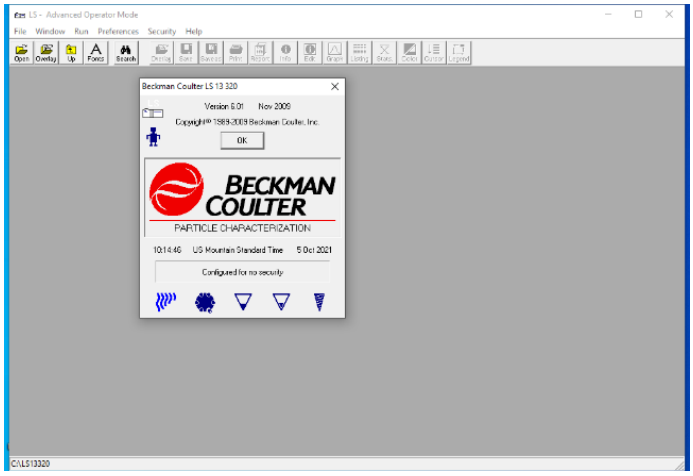


10. Running the Particle Size: After 48 hours preparation process, the samples are ready to be run on the laser diffraction machine.

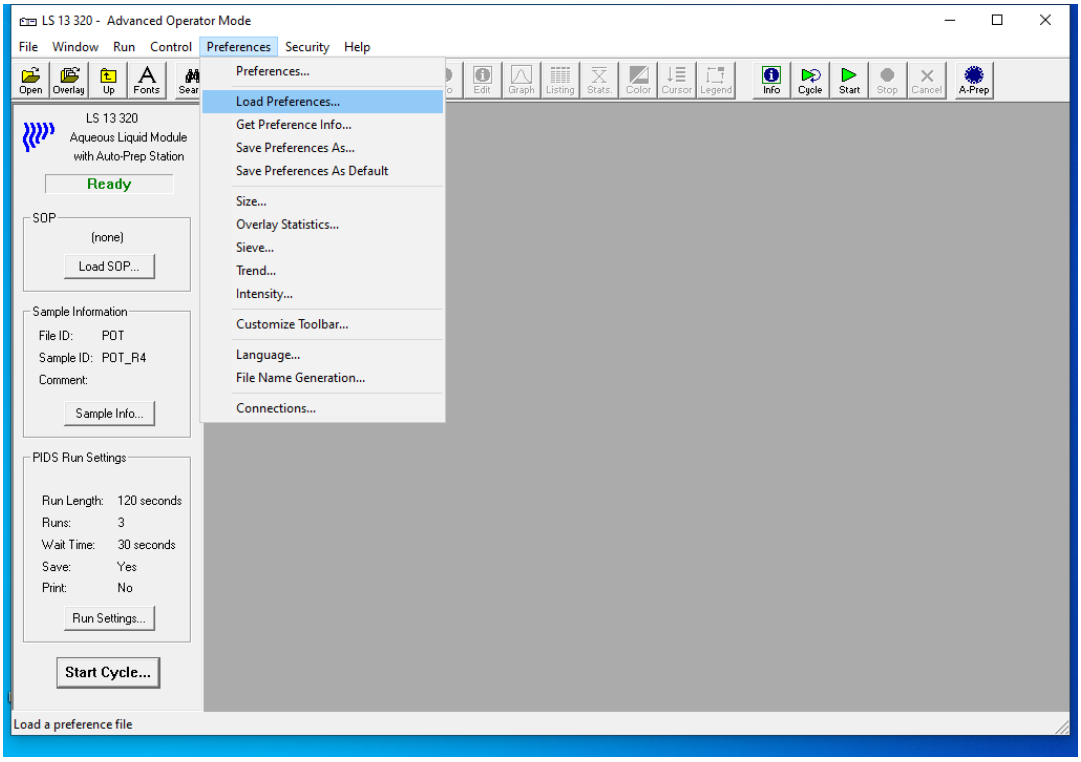
- Turn on the fan located on the back of the Aqueous Liquid Module (The power switch is located under the bottle of dispersant on the back right of the machine).

- Pick up the sonicator and slide it onto the mount located above the water dish.

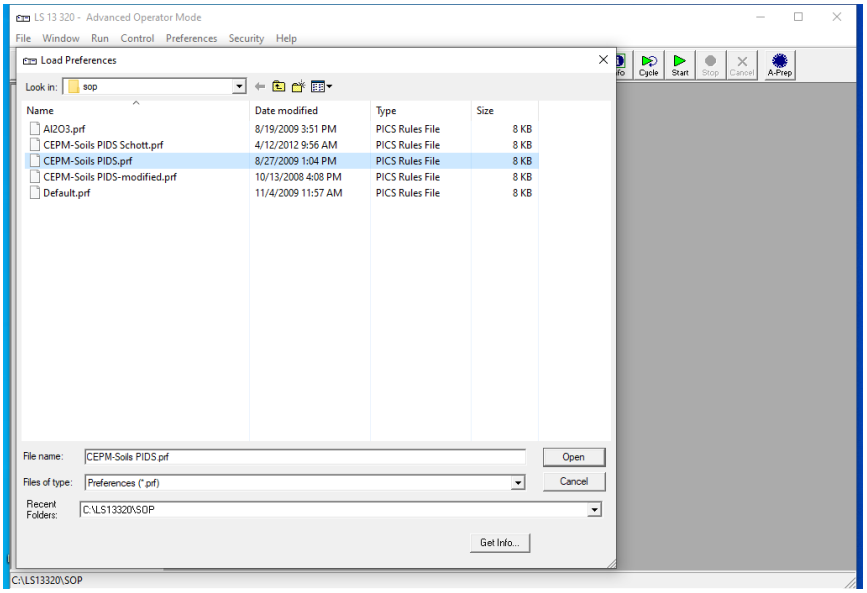
- Log into the computer using the password “1980vanGenuchten” on the ”Lab” account.
- Open the software on the computer.



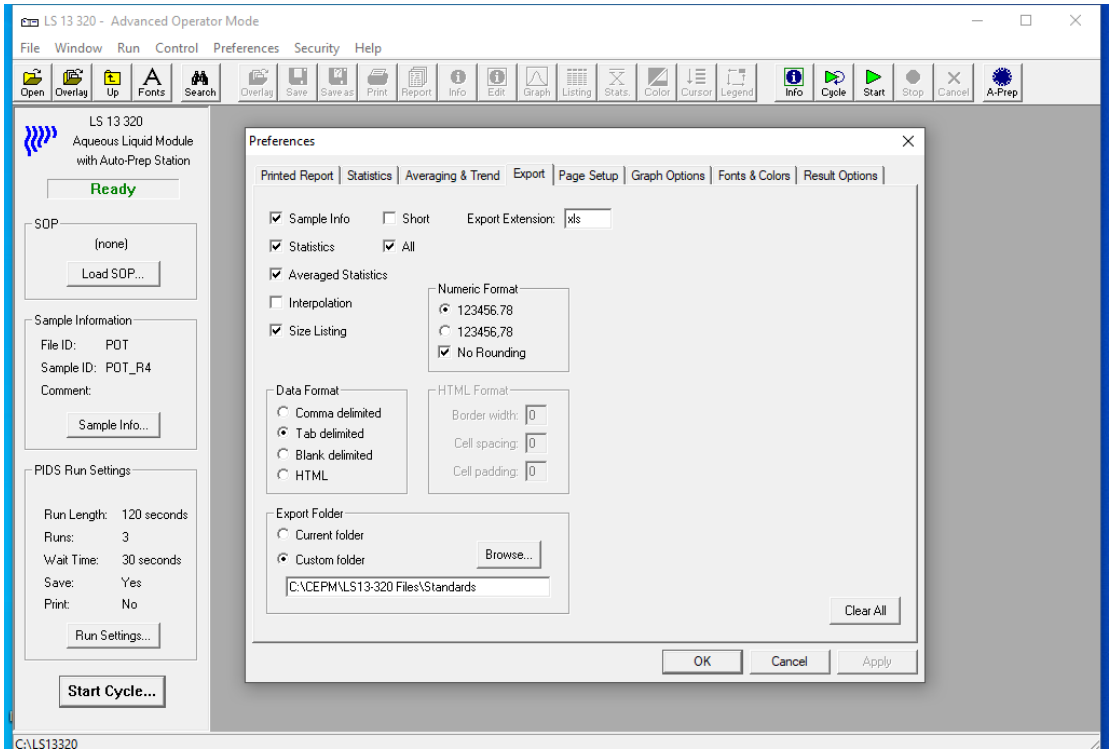
- Select “Preference” and the “Load Preferences”.



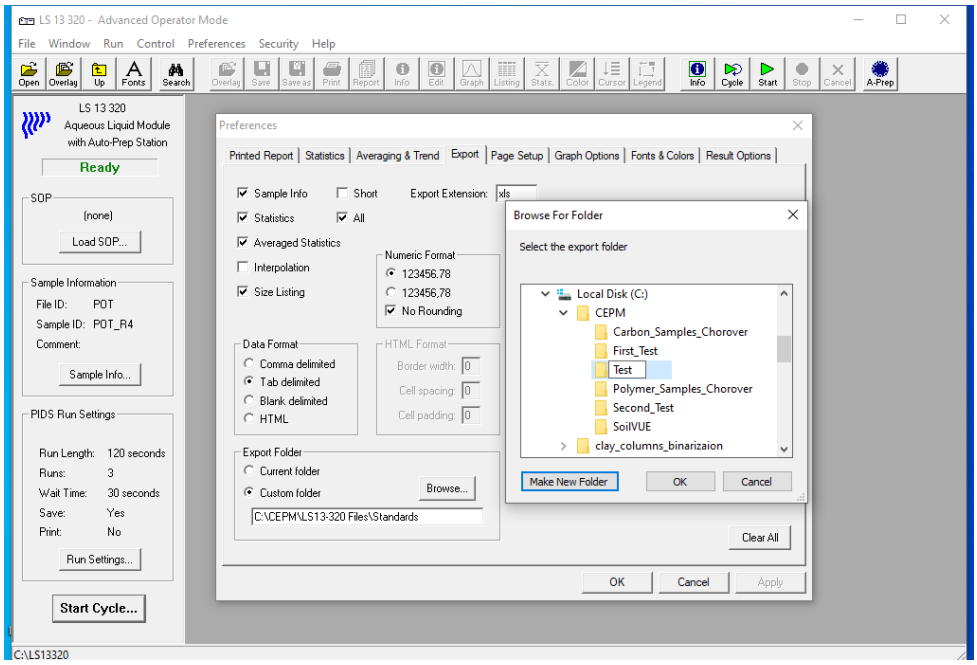
- Select the PIDS preference (the unmodified version). Click “Open” to apply the setting.



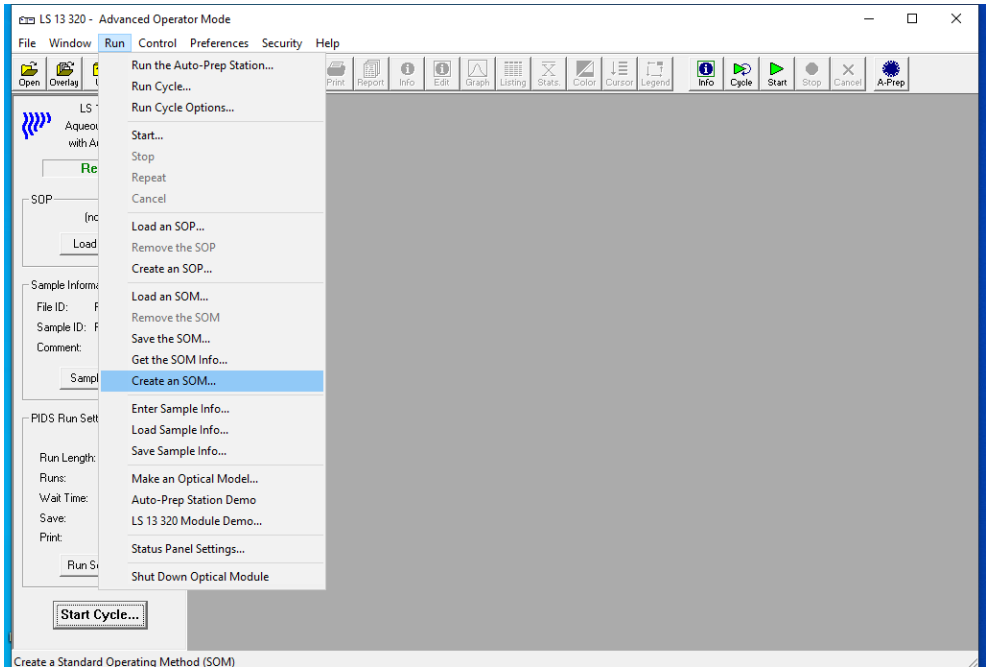
- Select “Preferences” a second time, and then select “Preferences”. Go to the “Export” tab and hit “Browse” at the bottom of the box. This allows us to create a folder to export it.



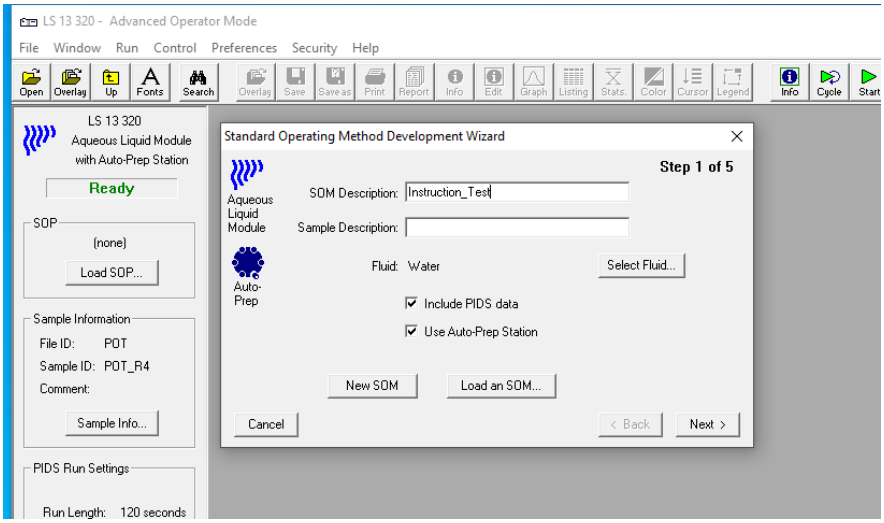
- Under the CEPM folder in the C Drive create a folder with the project name. all the data that is created by the software will be stored here.



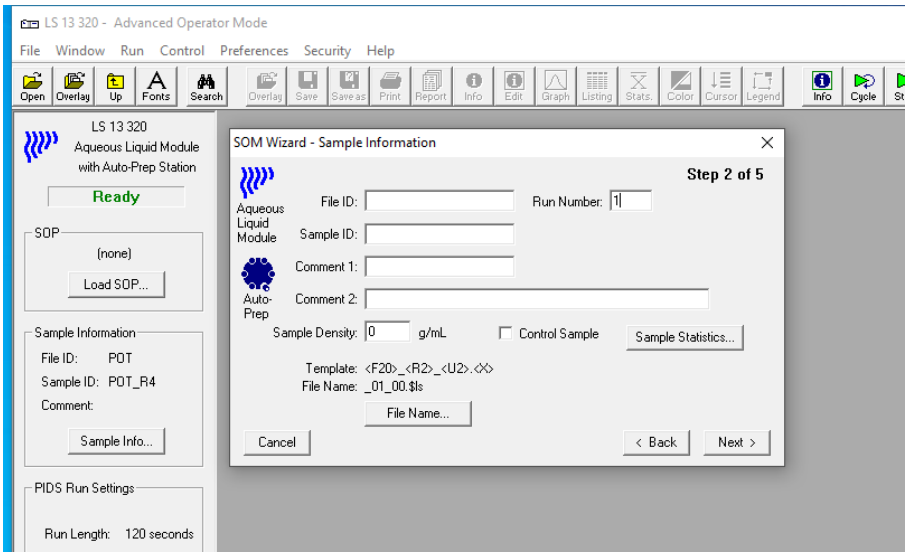
- Go to the “Run” on the toolbar. Select “Create an SOM”. This will create a standard operating method for the samples. There are five pages that the operating method setup goes through.

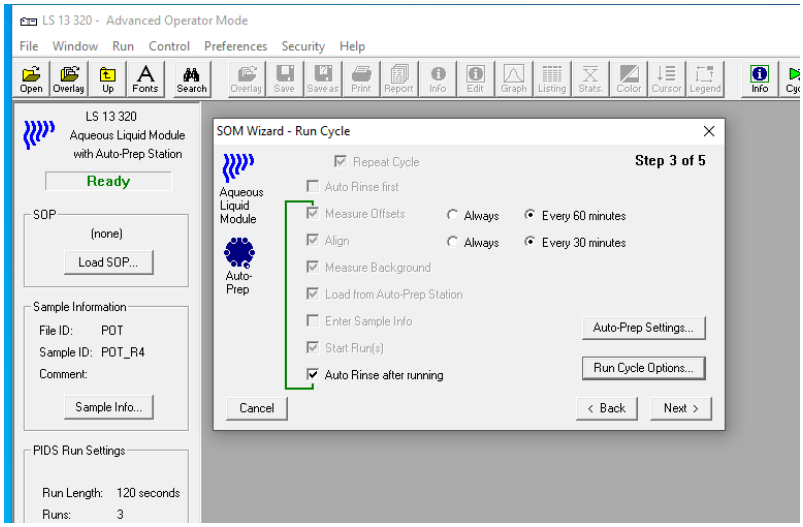


- On the first page erase the sample name that the software generates.

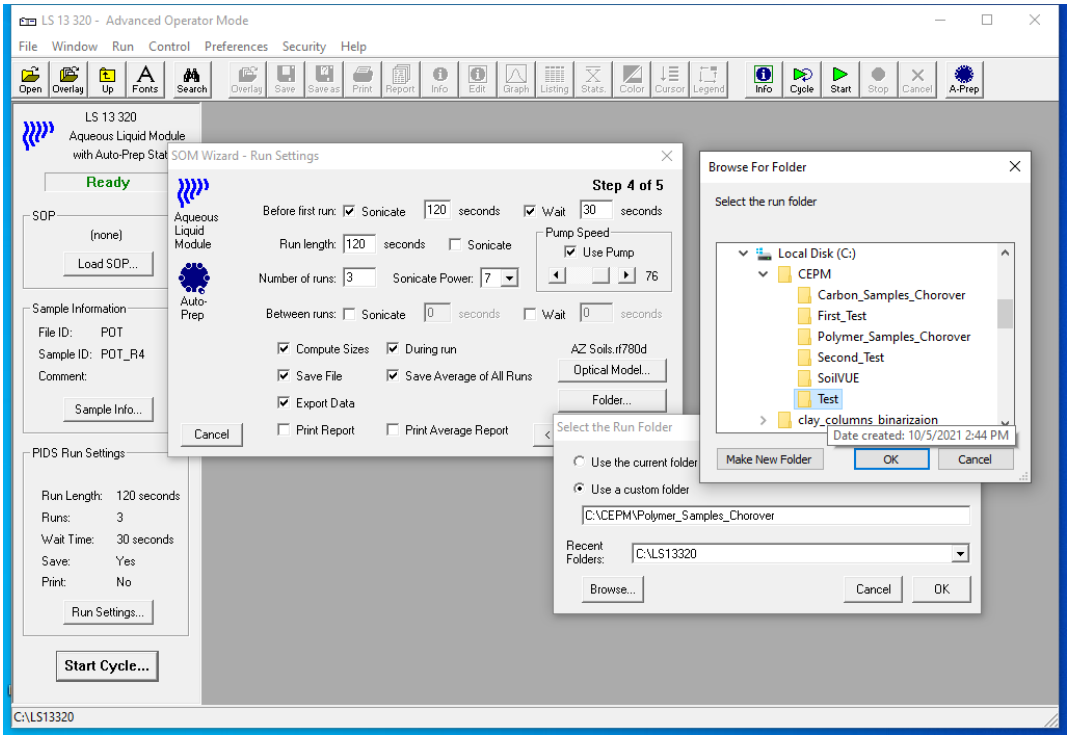


- On pages two and three do not require any input on your behalf.

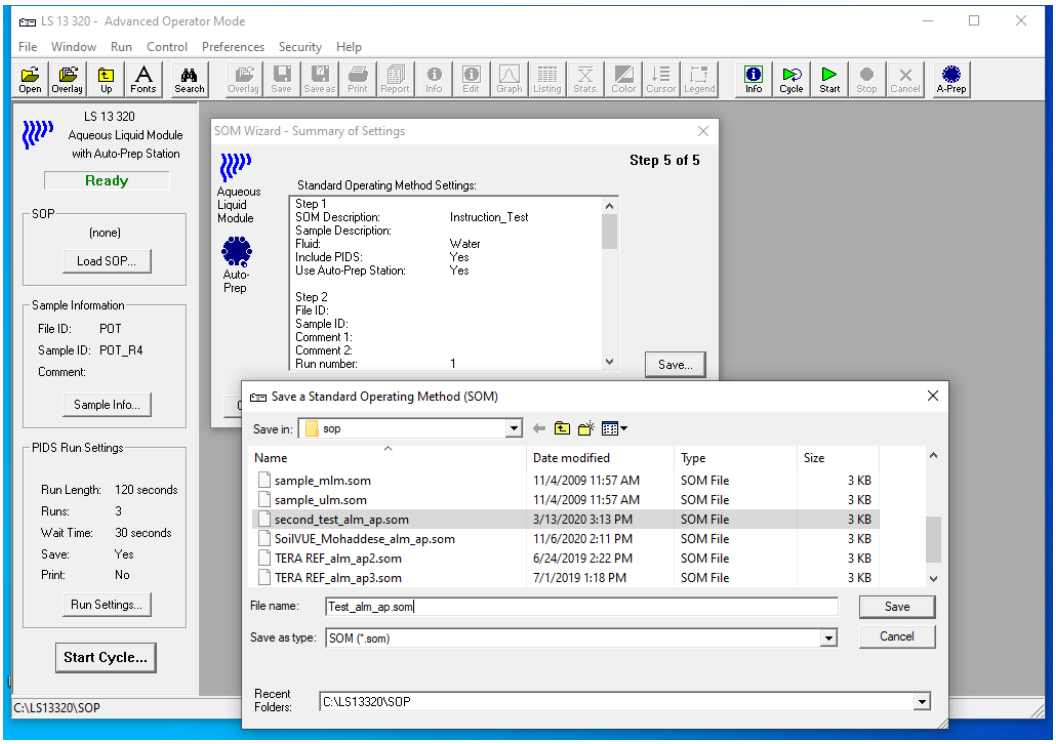




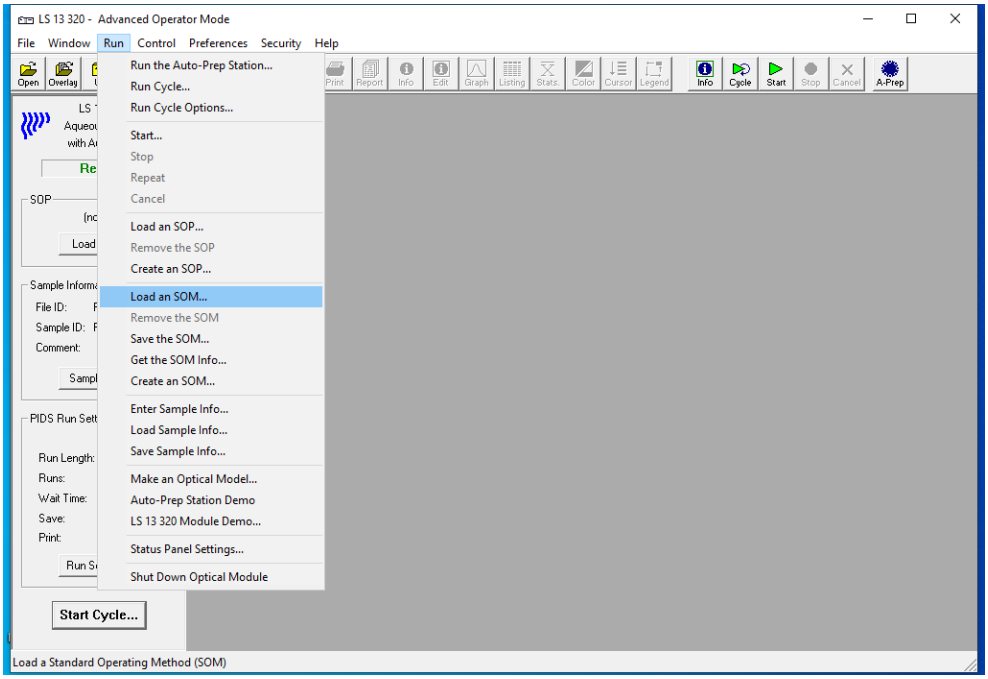
- On page four, select “Folder”. Here chose the folder that was created in the “Preferences” section. This will ensure that the data will be stored there.

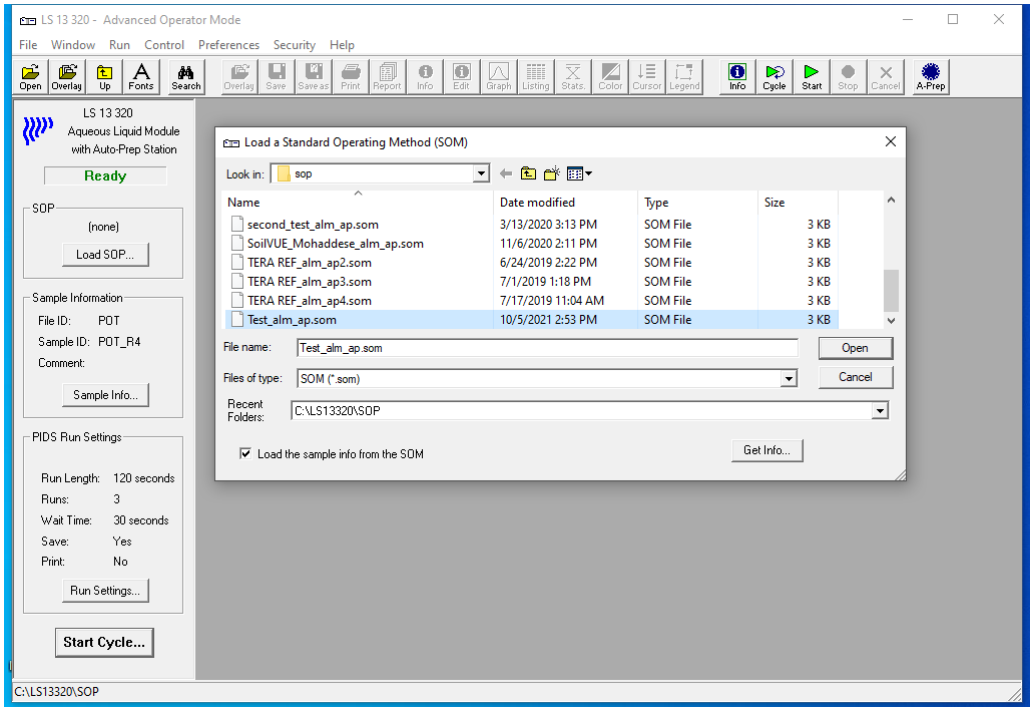


- On page five save the SOM. The title for the SOM is traditionally saved CEPM Soils PIDS 10-14-21 (changed to whatever the date of run is). Save and exit the SOM setup.

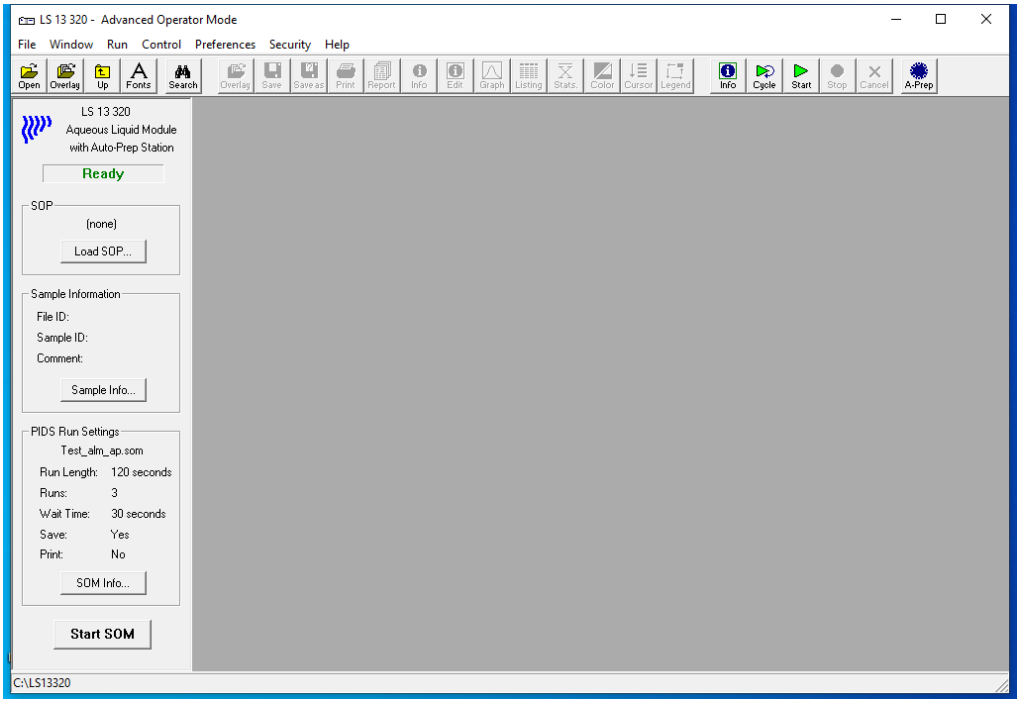


- Load the SOM by going to the “Run” and selecting “Load and SOM”. Find the SOM you just created using the “CEPM Soils PIDS…” and open the file.





- In the corner of the software select “Start SOM”. It will bring you to page where you will type in all the sample names that correspond to the test tubes to be loaded. Nothing needs to be typed in the Comments/Notes section



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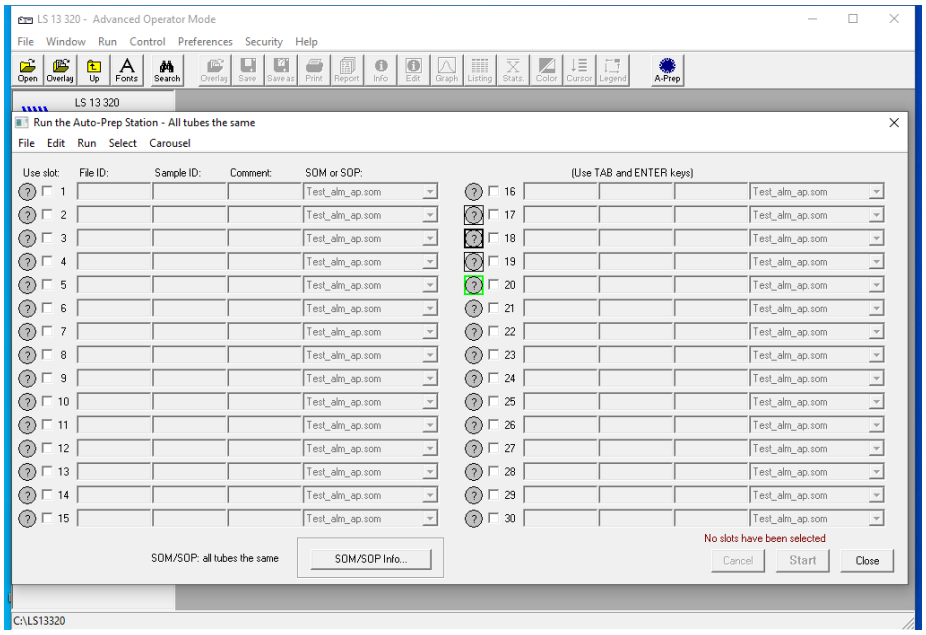
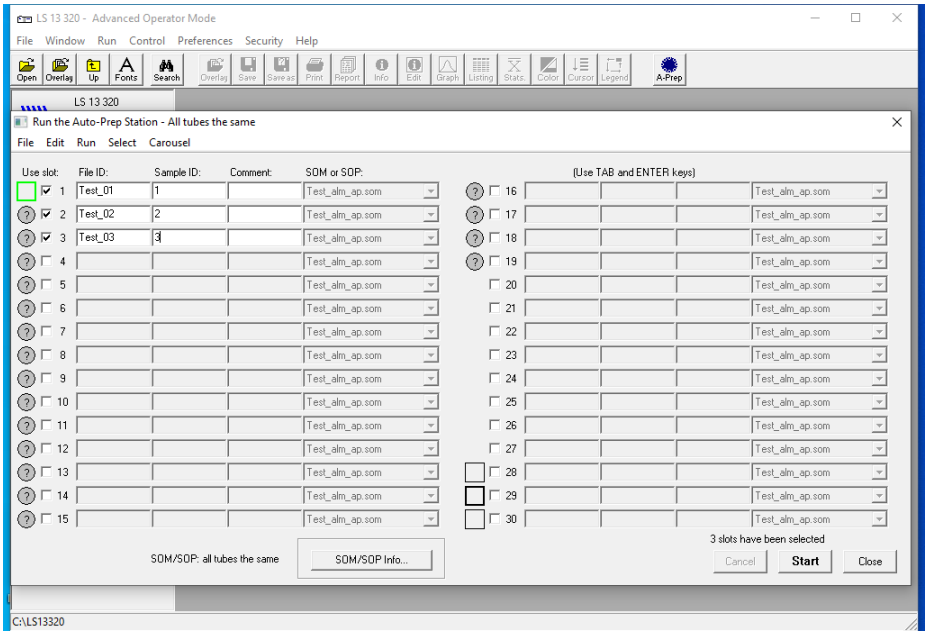
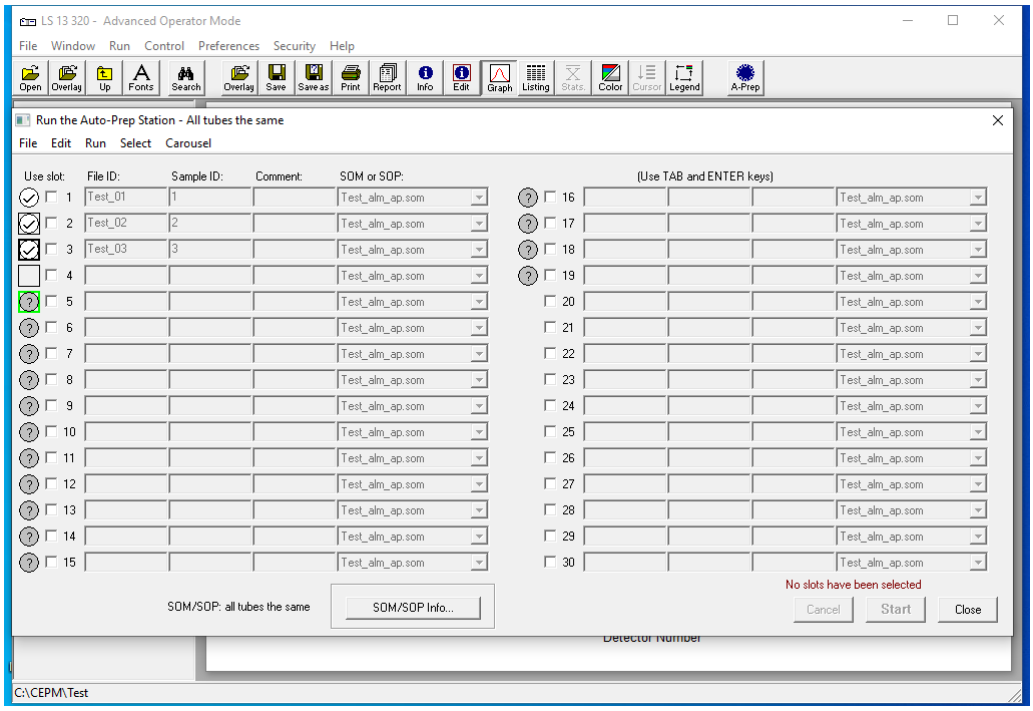




- Before you hit “Start”, after typing in all the names, load the tubes onto the machine. Be sure that each test tube has a piece of white tape sticking on the back, and this tape is facing the outside of the carousel. The machine has a camera that looks for next sample and it has a hard time noticing the test tube without the tape.

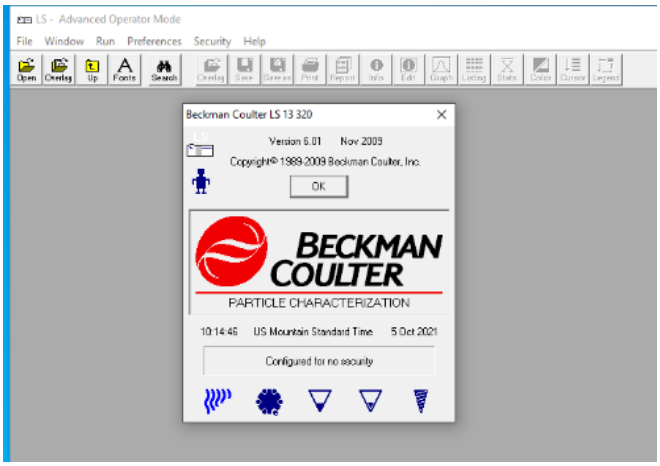


- Bring the gray box with sample names to the bottom of the screen so that the white (main) screen can be viewed. The carousel automatically turns to the first sample and the process should begin.

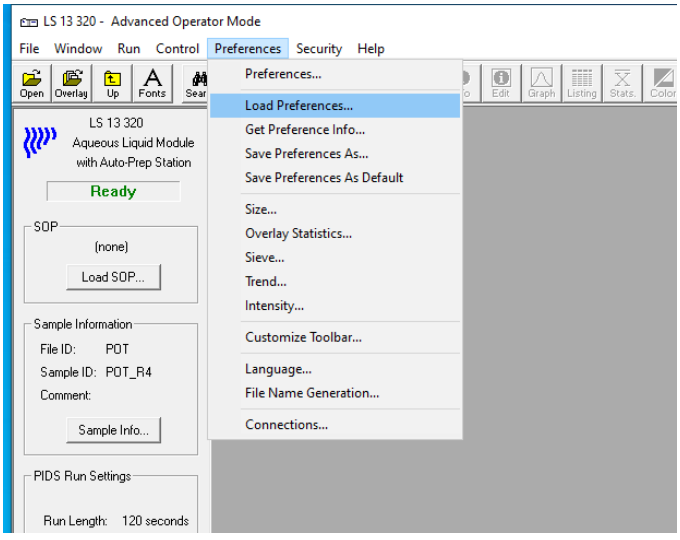
11. **Closing the Software:** once all the samples have been run through the laser diffraction software close the gray box that has samples name on it. Then select “Remove the SOM”. Go back to the “Start Cycle” button and unclick all of the boxes except for one “Auto Rinse”. Make sure that “Repeat Cycle” is also not selected.

- Once the “Auto Rinse” is completed select “Control” and “Move Carousel to Home” this allows for access to all of tubes which can then be removed and washed. Then go to the “Control” and select “Pump Off”. Once the pump has shut down the software can be exited and then the fan (in the back) can be turned off.

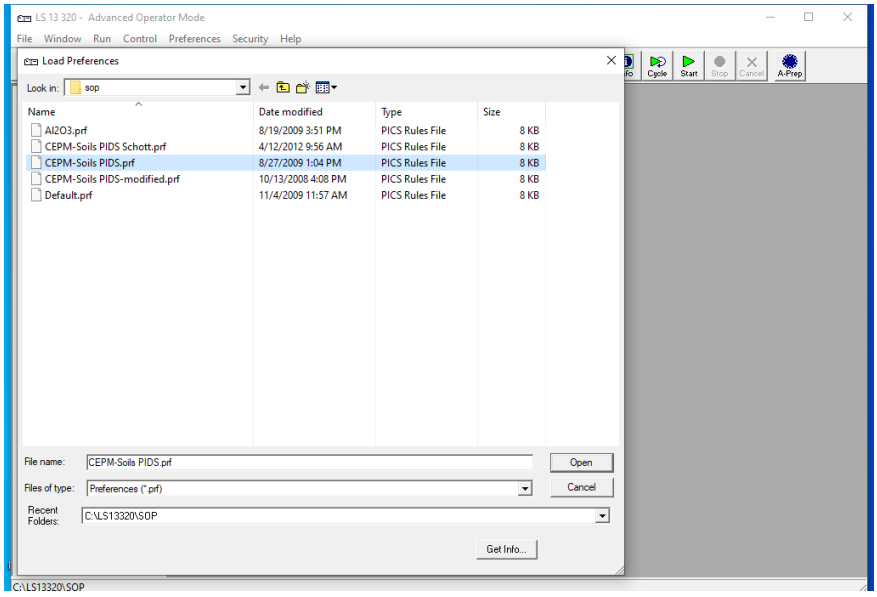
12. **Create Average files in Excel Format:** Open the software on the computer. Hit “OK” to exit to the main screen.



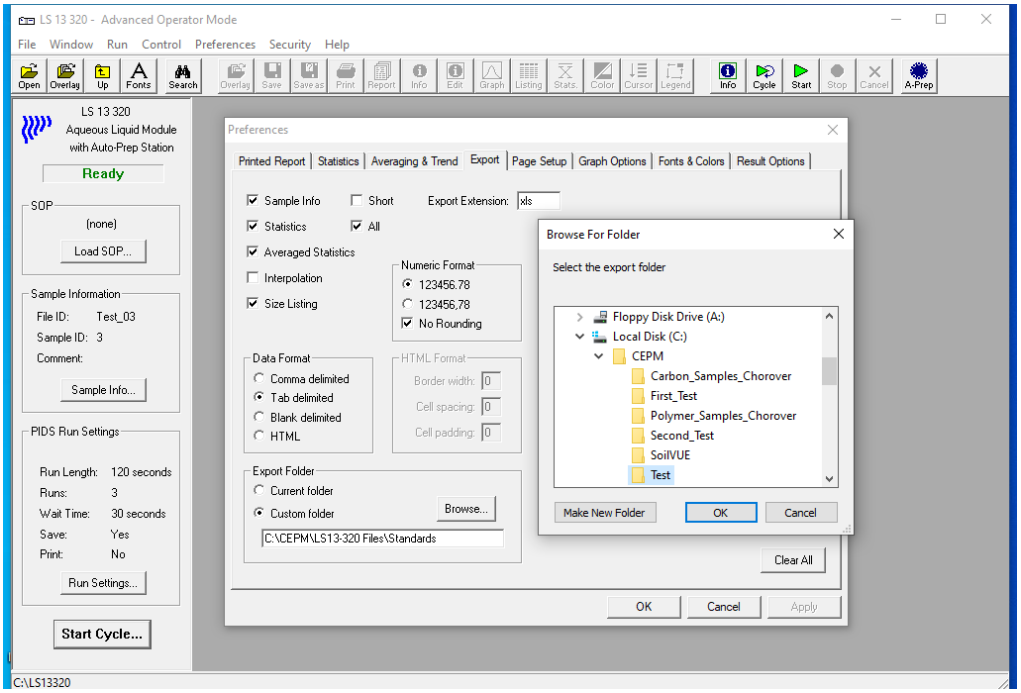
- Select “Preference” and the “Load Preferences”.



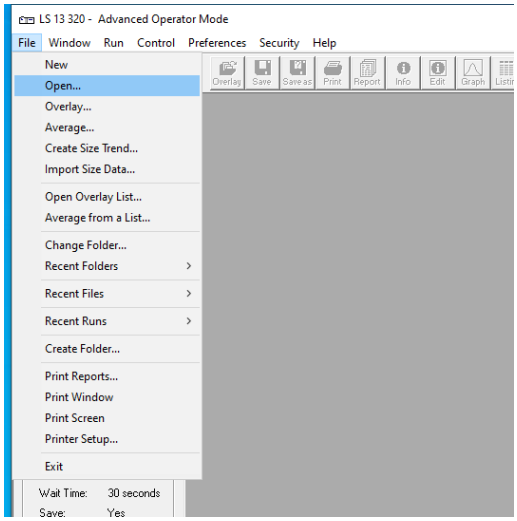
- Select the PIDS preference (the unmodified version). Click “Open” to apply the setting.



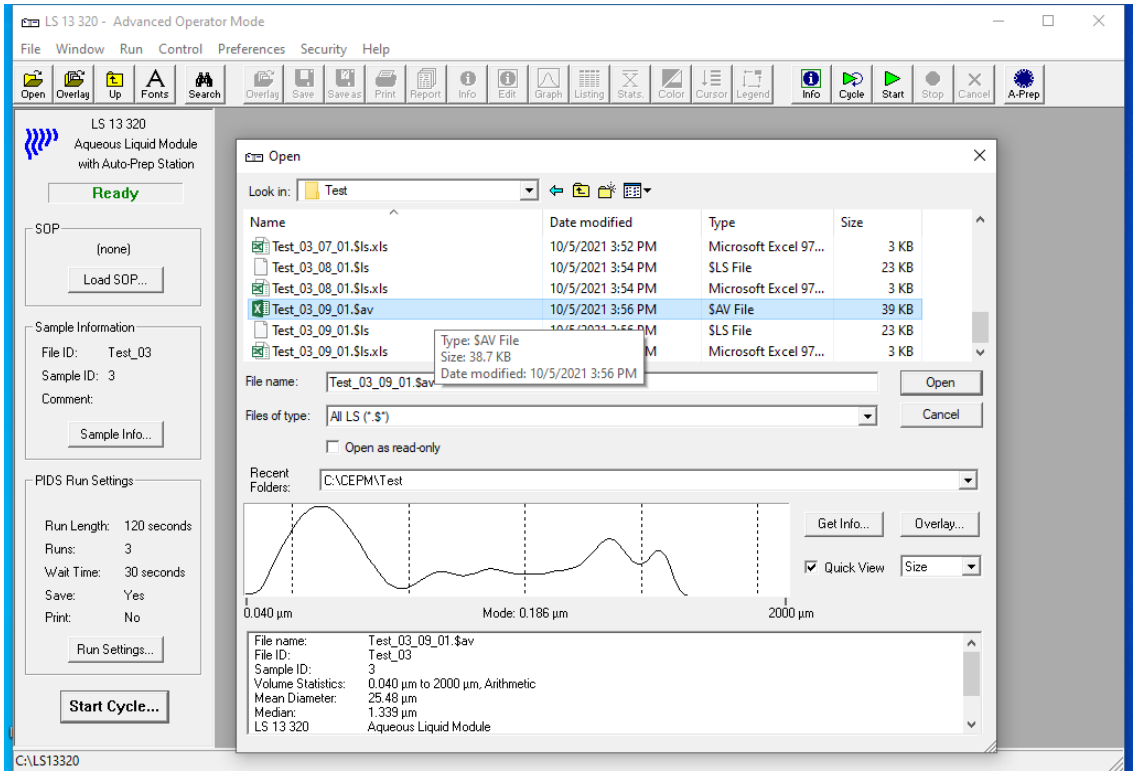
- Select “Preferences” a second time, and then select “Preferences”. Click on the “Export” tab to designate the export folder. In the “Export Folder” box, leave the “Custom Folder” bullet checked. Select “Browse”. Select the folder that currently contains all the run files for this project. Then hit “Apply” and “OK” to confirm the changes and exit out of the “Preferences” box.



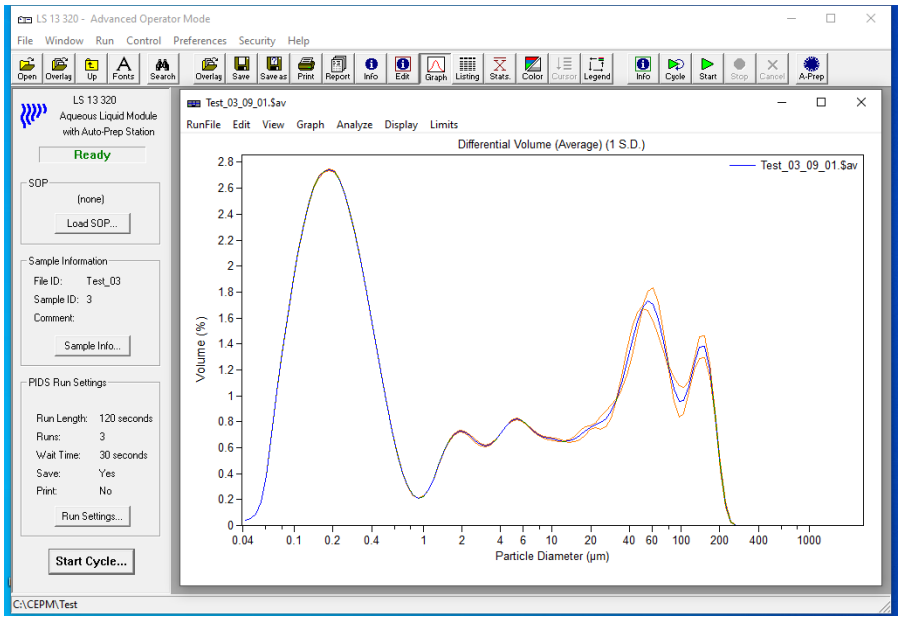
- Select “open” at the top left corner of the screen.



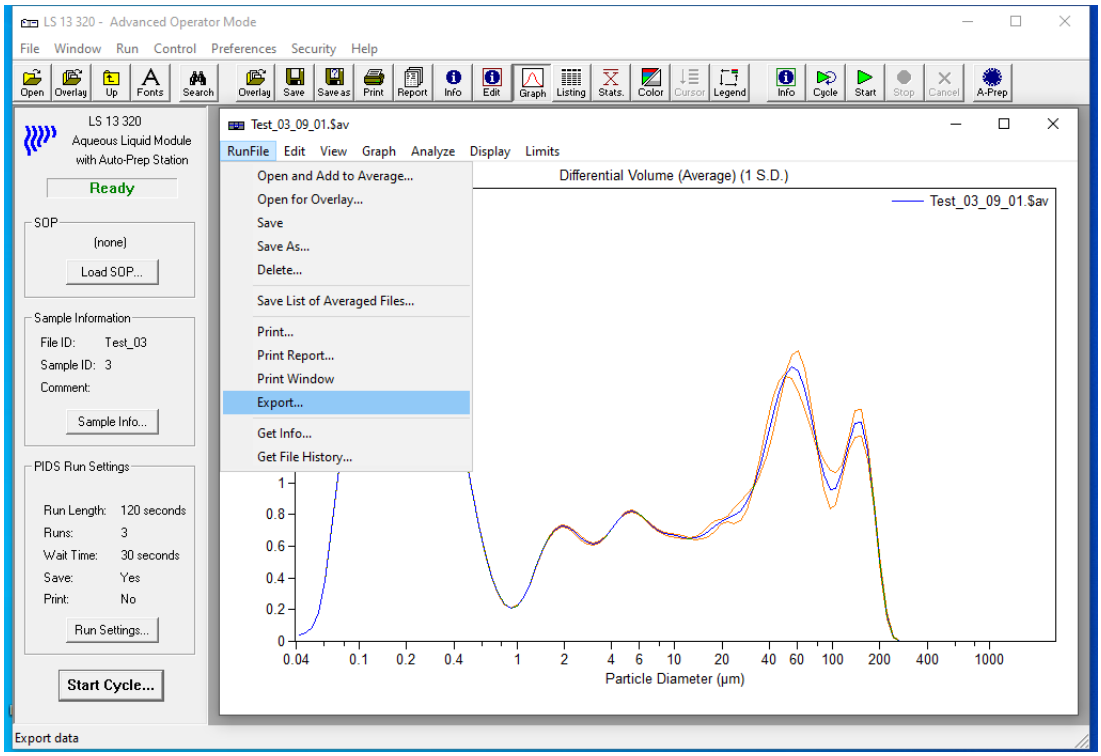
- From there find the folder that all the run files are saved. Open it and select the first samples’ average file. The icon next to the file will be blank page and the file name will end in ”. $av”.



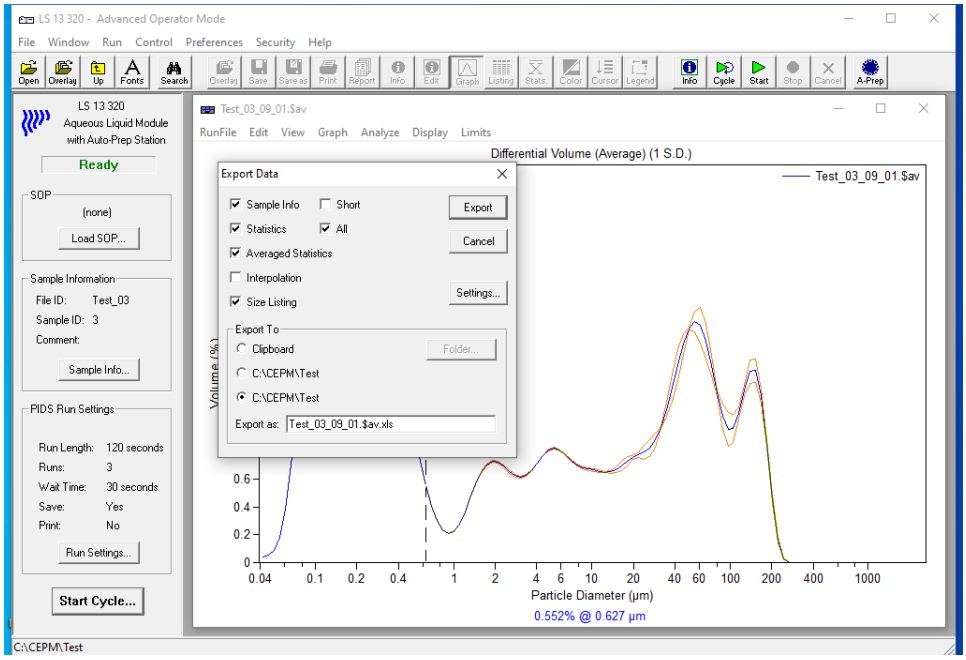
- Select “Open” and the first run’s curve will appear. The curve should be smoothed and contain three thinly colored lines.

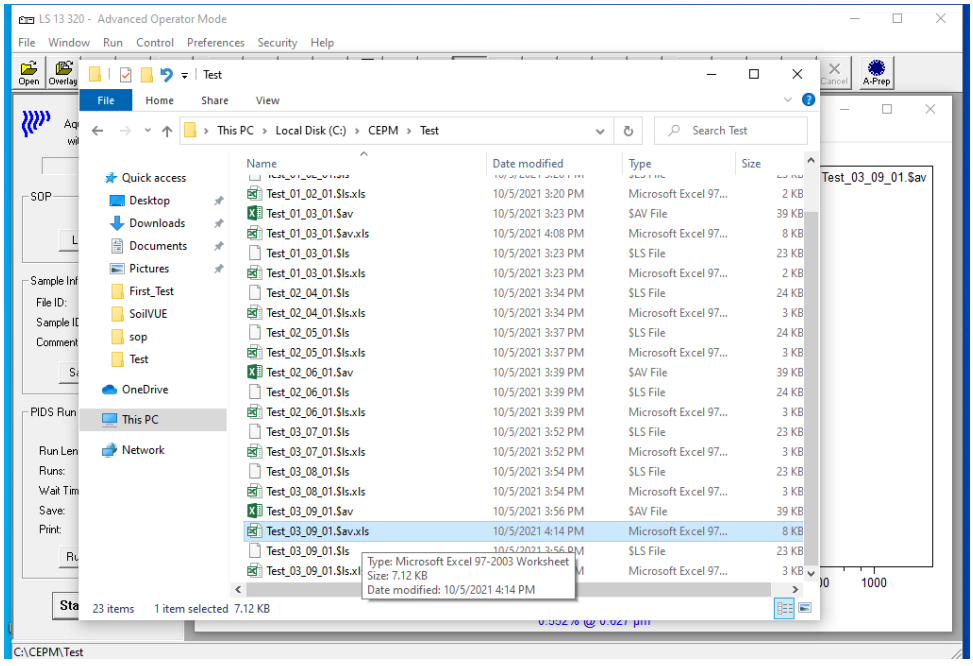


- Select “RunFile” in the upper left-hand corner and select “Export…” from the drop-down list.



- Check the box that says, “Size Listing” and then click “Export”. The graph of the sample can now be exited, and the second sample can be selected. Continue until all the samples have been exported.





- Once all the average files have been exported to an Excel format, the software can be exited. Only Excel spreadsheets will be used for the rest of the project.

- Copy and paste data from the Excel files generated in the previous step to the appropriate locations in the provided template Excel file.